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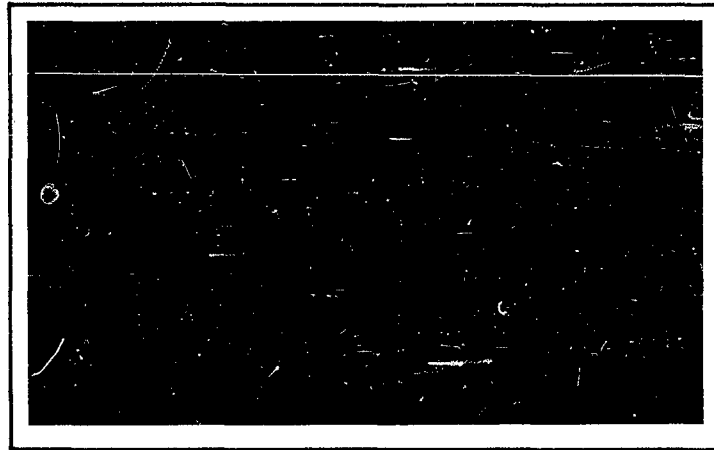
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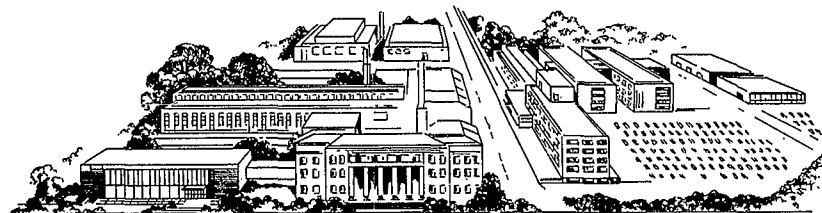
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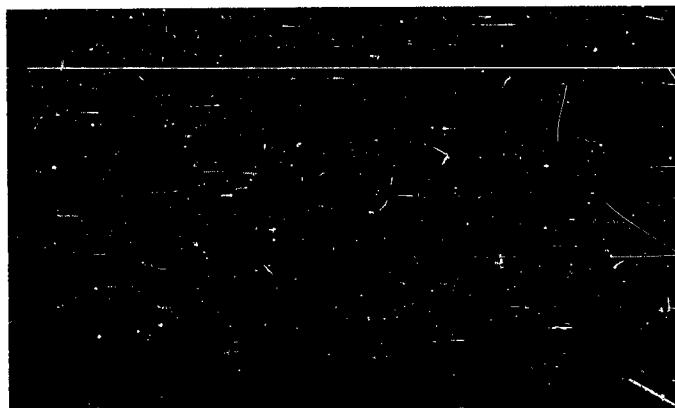


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FIRST QUARTERLY PROGRESS REPORT

on

FURTHER DEVELOPMENT OF DUCTILE
TUNGSTEN-BASE SHEET ALLOY

to

BUREAU OF NAVAL WEAPONS

May 8, 1963

J. L. Ratliff, D. J. Maykuth, H. R. Ogden, and R. I. Jaffee

Prepared under Navy
Bureau of Naval Weapons
Contract N600(19)-59738

For the Period: February 8, 1963 - May 8, 1963

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An additional 38 copies have been sent directly to the organizations listed on the distribution list provided by your office.

Very truly yours,

ORIGINAL SIGNED BY

H. R. OGDEN

H. R. Ogden, Chief
Nonferrous Metallurgy Division

(DJM)
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ABSTRACT

Ternary alloys containing 5 per cent rhenium in combination with ThO_2 inert oxide dispersions were prepared to determine the limits of fabricability for oxide contents between 4 and 7 volume per cent. Results from this initial work indicated that good workability persists to at least the 5 volume per cent level of either ThO_2 or ZrO_2 .

A group of doped but otherwise unalloyed tungsten powders was prepared to evaluate the effect of alkali silicate additives on the recrystallization behavior of tungsten sheet.

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FURTHER DEVELOPMENT OF DUCTILE TUNGSTEN-BASE SHEET ALLOY

by

J. L. Ratliff, D. J. Maykuth, H. R. Ogden, and R. I. Jaffee

INTRODUCTION

This is the First Quarterly Progress Report on the "Further Development of a Ductile Tungsten-Base Sheet Alloy" which describes the results of research performed over the period February 8, 1963, through May 8, 1963.

The primary objective of this program is to develop a tungsten-base alloy having excellent fabricability and a ductile-to-brittle transition temperature below room temperature. Aside from maintaining high elevated-temperatures strengths, secondary objectives are to develop alloys with high recrystallization temperatures and improved recrystallized structures.

This research continues the effort initiated under Contract NOW61-0677-C where both the separate and combined effects of inert dispersed oxides and high-valency metal additions on the properties of tungsten sheet were first explored.⁽¹⁾ Of various metallic additions, a 5 per cent addition of rhenium to tungsten was found most effective in decreasing the bend transition temperature and increasing the recrystallization temperature of tungsten. Incorporating 4 volume per cent of thoria in this base resulted in further favorable property advantages. Combinations of 8 volume per cent of thoria with 1 per cent osmium or 0.3 per cent iridium showed similar benefits although these alloys were more difficult to fabricate.

In the present program, the experimental approach is to optimize the composition and fabrication procedure for alloys incorporating 5 per cent rhenium plus the maximum amount of thoria or zirconia commensurate with good workability. A study of the effectiveness of various auxiliary doping additions in further controlling grain-growth characteristics is believed to constitute an important part of this alloy development.

SUMMARY

Powder-metallurgical preparation of the solid solution-dispersion and doped alloys was initiated. The solid solution-dispersion or ternary materials contained from 4 to 7 volume per cent of either ThO_2 or ZrO_2 added aqueously in combination with 5 per cent rhenium. Four doped compositions, each prepared in duplicate, constituted the group of doped materials. Three of the alloys in this group were prepared from soluble-salt additions to unalloyed powder while the fourth consisted of a commercially doped grade of tungsten powder (Type 218). Common dopants for the series of doped compositions included K_2O , SiO_2 , and Al_2O_3 .

After drying and conditioning, each alloy powder charge was compacted by mechanical die pressing into $1/4 \times 1/2 \times 7$ -inch bars. All of the ternary alloys were pressed at 50,000 psi while the duplicate compositions of the doped alloys were pressed at both 30,000 psi and 50,000 psi. Subsequent sintering of both groups of materials was accomplished without difficulty; however, for the solid solution-dispersion group, ThO_2 impeded the sintering process more than ZrO_2 .

The effect of variable pressing pressure applied to the doped alloys was reflected in the sintering behaviors of the two most heavily doped compositions. Specifically, the bars pressed at 30,000 psi rather than 50,000 psi showed improved densification on sintering.

Metallographic examination revealed that some rhenium-rich areas persisted in each of the as-sintered bars to which rhenium was added. Also, some tendency toward ThO_2 agglomeration was observed. The probable causes for these structural deficiencies were determined and corrective measures are being taken to avoid these difficulties in the future.

Rolling experiments with these preliminary bars were not conclusive. However, the results indicated that good workability in the W-5Re base composition persisted to levels of at least 5 volume per cent of ThO_2 and ZrO_2 .

MATERIALS

Table 1 lists the suppliers, particle sizes, and purities of the tungsten and rhenium powders purchased for use in this program. In addition, a supply of Type 218 tungsten powder was also procured from the General Electric Company for evaluation in this work. This material is a proprietary, alkali-silicate-doped powder which is used extensively in the manufacture of nonsag filament wire. As noted in the literature⁽²⁾, this material is prepared by adding small amounts of potassium silicate and AlCl_3 in aqueous solution to tungsten oxide powder which is subsequently dried and reduced to metal with hydrogen.

TABLE 1. SUMMARY OF SUPPLIERS, PARTICLE SIZES, AND PURITIES OF METAL-POWDER ADDITIONS

Material	Supplier	Average Particle Size, microns	Nominal Purity, wt. %	Analysis, ppm									
				O	C	Mo	Cu	Ni	Fe	Ca	K	Si	Al
Unalloyed tungsten	General Electric	2.31	99.93	422	24	100	<3	17	50	4	28	<7	<6
Rhenium	Chase Brass	<74 ^(a)	99.99	--	--	--	<1	--	16	<1	--	<1	<1

(a) <74 designates powder of -200 mesh size.

The principal oxides of interest to this program for use as dispersion and doping additions are ThO_2 , ZrO_2 , Al_2O_3 , K_2O , and SiO_2 . The thorium and zirconia additions were especially selected for dispersion applications because of their excellent thermodynamic stability in tungsten at temperatures approaching its melting point, 3410 C.

Accordingly, supplies of high-purity water-soluble $\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}$ and a 0.01-micron ZrO_2 sol were obtained, thus enabling the preparation of both the ThO_2 and ZrO_2 dispersion alloys by aqueous methods.

The oxides of aluminum, potassium, and silicon were selected for combination with unalloyed tungsten powder because of their known ability as doping additions. (2,3) Accordingly, small quantities of water-soluble salts of these elements, AlCl_3 and $\text{K}_2\text{Si}_4\text{O}_9$, were obtained, thus also enabling the aqueous preparation of alloys doped from these additions.

ALLOY PREPARATION

Two groups of tungsten-base alloys, listed in Table 2 with their respective alloy compositions, were prepared for initial investigations.

The first group consisted of a series of ternary alloys containing 5 per cent rhenium in combination with either thorium or zirconia in amounts from 4 through 7 volume per cent. These alloys were prepared to define the upper limit of oxide content commensurate with good workability.

The second group consisted of a series of doped, but otherwise unalloyed, tungsten samples. These were prepared to screen the Type 218 and other compositions to determine which would be most suitable as a base material for future W-Re-($\text{ThO}_2/\text{ZrO}_2$) alloys.

The doping levels for the three experimental compositions shown in Table 2 were selected after a review of the literature. As indicated, the compositions were selected to explore combined Al_2O_3 and SiO_2 additions at levels of 0.1 and 0.01 weight per cent and at $\text{Al}_2\text{O}_3:\text{SiO}_2$ ratios of 1:4 and 1:9, as well.

Mixing, Compacting, and Presintering

The aqueous preparation of dispersion and experimentally doped alloys was accomplished according to the following procedure:

- (1) Prepare alloy powder charges by adding appropriate quantities of water-soluble salts and metal powder materials to 40 milliliters of distilled water, forming a slurry.
- (2) Dry each slurry by evaporation on a hot plate while continuously stirring to homogenize the mixture.
- (3) Condition the dried and agglomerated powder charges by pulverizing, baking for 2 hours at 600 C in a dry hydrogen atmosphere, screening through -325 mesh and cone blending for 1 hour prior to compaction.
- (4) Compact the powder charges by mechanical die pressing into 1/4 x 1/2 x 7-inch size bars.

TABLE 2. SUMMARY OF ALLOY COMPOSITIONS, SINTERING CONDITIONS, AND DENSITIES

Alloy	Nominal Alloy Content		Compacting Pressure, ksi	Sintering Conditions		Density		
	Oxide Volume Per Cent	Weight Per Cent		Time, hr	Temperature, C	Theoretical (a), g/cm ³	Per Cent of Theoretical Initial(b) Final	
W-1	--	100W	50	2	2600	19.30	58	94.3
W-2	--	100W	30	2	2600	19.30	53	93.8
Unalloyed Tungsten								
WT-1	4ThO ₂	W-5Re-2, 2ThO ₂	50	2	2600	19.00	54	88
WT-2	5ThO ₂	W-5Re-2, 7ThO ₂	50	2	2600	18.90	88	92.4
WT-3	6ThO ₂	W-5Re-3, 2ThO ₂	50	4	2600	18.90	49	93.3
WT-4	7ThO ₂	W-5Re-3, 8ThO ₂	50	4	2600	18.82	51	91.0
WT-5	4ZrO ₂	W-5Re-1, 2ZrO ₂	50	4	2600	18.72	53	90.0
WT-6	5ZrO ₂	W-5Re-1, 5ZrO ₂	50	4	2600	18.82	55	95.4
WT-7	6ZrO ₂	W-5Re-1, 8ZrO ₂	50	4	2600	18.69	57	96.7
WT-8	7ZrO ₂	W-5Re-2, 1ZrO ₂	50	4	2600	18.55	58	96.8
						18.41	58	95.8
Solid Solution-Dispersion Alloys								
Doped Alloys								
WD-1	--	Type 218	30	4	2600	19.30	57	90.0
WD-2	--	Type 218	50	4	2600	19.30	62	89
WD-3	--	W-0.02Al ₂ O ₃ -0.08SiO ₂ -0.032K ₂ O	30	2	2600	19.30	89	90.2
WD-4	--	W-0.02Al ₂ O ₃ -0.08SiO ₂ -0.032K ₂ O	50	2	2600	19.30	54	89
WD-5	--	W-0.002Al ₂ O ₃ -0.008SiO ₂ -0.0032K ₂ O	30	4	2600	19.30	89	94.8
WD-6	--	W-0.002Al ₂ O ₃ -0.008SiO ₂ -0.0032K ₂ O	50	4	2600	19.30	58	93.6
WD-7	--	W-0.001Al ₂ O ₃ -0.009SiO ₂ -0.0036K ₂ O	30	4	2600	19.30	58	94.1
WD-8	--	W-0.001Al ₂ O ₃ -0.009SiO ₂ -0.0036K ₂ O	50	4	2600	19.30	59	95.1
						19.30	53	94.8
						19.30	59	

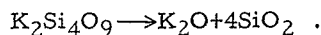
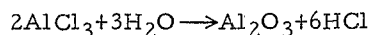
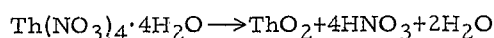
(a) Calculated using the relationship $\frac{1}{d} = \frac{Wt\%}{100 d_A} + \frac{Wt\%}{100 d_B} + \dots$, where d values for ThO₂, ZrO₂, W, and Re were taken as 10.03, 5.6, 19.30, and 21.04.

(b) After presintering for 4 hours at 1200 C in dry hydrogen.

- (5) Presinter each compacted bar for 4 hours at 1200 C in a dry hydrogen atmosphere.

With respect to the solid solution-dispersion alloys, an additional precautionary measure was employed in the first step of the above procedure to insure the initial preparation of homogeneous W-5Re powder mixtures. Beginning with the rhenium powder, minus 325-mesh fines were screened from the commercial powder lot which as-produced was only minus 200-mesh. The fines were then cone blended for 5 hours during which time unalloyed tungsten powder was added in increasing amounts until the desired compositional balance was achieved. The soluble-salt content was then added and the remainder of the processing completed as usual.

The material for each powder bar was calculated to yield a total mass of 160 grains, assuming that the soluble-salt content would decompose thermally during precompaction baking and presintering to form oxides in accordance with the following reactions:



Both precompaction baking and presintering are regarded as necessary steps to the subsequent production of high quality bars. The former serves to decompose the majority of the soluble-salt content in the loose-powder charges, while the latter completes the decomposition reactions and imparts sufficient green strength to the powder bars to permit handling prior to sintering.

Consolidation of the alloys was carried out by mechanically pressing each loose-powder charge into 1/4 x 1/2 x 7-inch bars. Using a loading rate of 0.10 inches per minute, a common compacting pressure of 50,000 psi was applied to all of the W-5Re-base alloys, which resulted in presintered densities ranging between 49 and 58 per cent of theoretical.

Compacting pressures of 50,000 and 30,000 psi were applied to the four doped compositions (as well as to the duplicate bars of unalloyed tungsten) to explore the effect of this variable on subsequent sintering behavior. Generally, these differences in initial consolidation practices were reflected in each of the alloys after presintering, as consistently higher density was obtained for the 50,000-psi loading condition than for the 30,000-psi condition; i. e., densities ranged between 58 and 62 per cent and 53 and 58 per cent for the two conditions, respectively.

Sintering

Each of the alloys, including the unalloyed tungsten controls, was sintered by self-resistance heating in vacuum.

Sintering temperatures were measured optically by sighting on the center surface of each bar. True temperatures, T, were determined by the relationship

$$T = S + G + \Delta T$$

where S is the apparent surface temperature (optical pyrometer value), G is the glass correction, and ΔT is the black-body temperature correction for tungsten using a wavelength of 0.655 micron.

Sintering was effected by heating each bar to 1200 C, outgassing for 1 hour, then heating to 2600 C over a period of 4 hours and holding at this temperature for the time required to achieve the minimum target density of 90 per cent of theoretical. In this respect, sintering periods of 2, 4, and 4 to 6 hours were required to obtain or exceed the minimum standard density of unalloyed tungsten, the solid solution-dispersion, and the doped compositions, respectively.

Some general observations with respect to the sintering behaviors and trends in as-sintered densities (recorded in Table 2) for the solid solution-dispersion and doped alloys are presented as follows.

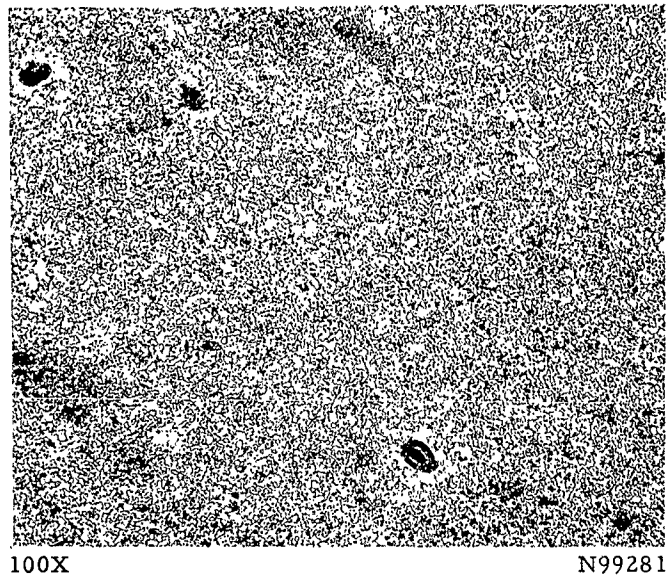
Comparing the behaviors of the solid solution-dispersion alloys, it was noted that W-5Re-ThO₂ alloys were more difficult to sinter to high densities than the W-5Re-ZrO₂ alloys. Thus, densities for the ThO₂ alloys ranged between 90.0 and 93.3 per cent, while values for the ZrO₂ alloys ranged between 95.4 and 96.8 per cent of theoretical. Also, for this group of materials, there was a slight indication that increasing amounts of ThO₂ from 4 volume per cent to 7 volume per cent impeded densification while identical levels of ZrO₂ had little or no effect on the ability of the alloys to densify.

One of the primary points concerning the sintering behaviors of the doped alloys was the question as to whether or not variations in compacting pressure would have any effect on the subsequent sintering behavior. As shown in Table 2, the use of a lower pressure tended to improve densification on sintering the Type 218 and the most heavily doped experimental alloy bars. Thus both of these compositions showed less resistance to sintering and sintered to higher densities after being pressed at 30,000 psi than when pressed at 50,000 psi. This behavior was especially true of the Type 218 powder as the bar pressed at 50,000 psi required 6 hours at 2600 C to attain a density of at least 90 per cent, whereas the same material pressed at 30,000 psi sintered to an acceptable density in only 4 hours at 2600 C.

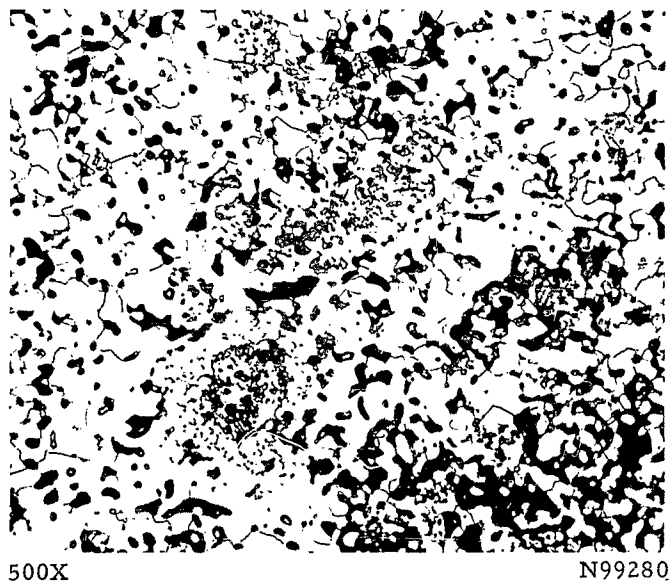
The other, more lightly doped compositions, behaved similarly to unalloyed tungsten in that subsequent sintered densities were, for the most part, independent of prior pressing conditions.

Slices cut from the ends of each of the sintered, solid-solution-dispersion alloy were examined metallographically to determine the relative effects of increasing amounts of ThO₂ and ZrO₂ dispersions on the resultant microstructures. Despite the initial care taken in preparing these alloys, the metallographic examinations revealed that all of these alloys contained some undissolved rhenium and/or rhenium-rich areas. Also, in the series of W-5Re-ThO₂ alloys, the normally random dispersion of ThO₂ was grossly agglomerated.

An example of the rhenium inhomogeneity is shown in Figure 1a for the W-5Re-4 volume per cent ThO₂ alloy. As noted earlier, the rhenium powder used to prepare these alloys was screened through -325 mesh, and thus contained rhenium particles up to 44 microns in diameter. Measurements on the sintered structures indicated that the observed rhenium-rich areas resulted from the fraction of the larger sized rhenium powders, i. e., those rhenium particles above about 20 microns in diameter were not



a.



b.

FIGURE 1. AS-SINTERED MICROSTRUCTURE ILLUSTRATING RHENIUM-RICH AREAS AND THORIA AGGLOMERATION IN THE W-5Re-4 VOL% ThO₂ ALLOY

Etchant: Murakami's reagent; structure in (a) oxidized anodically in 0.1 N H₃PO₄ solution.

taken into solution by the tungsten during sintering. The use of finer rhenium powder in the future preparation of these alloys is one obvious method which will be used to circumvent this difficulty.

The ThO₂ agglomeration in the sintered bars, illustrated in Figure 1b, apparently occurred as a result of insufficient mixing while evaporating the original slurries to dryness and/or inadequately blending the charges prior to compaction. Additional care and time in evaporating and blending the charges will be used to avoid this problem with future alloys.

Metallographic examination of the sintered structures of the doped alloy bars is now in process.

Fabrication

Despite their aforementioned inhomogeneities, each of the sintered alloy bars was screened for fabricability by flat-rolling to strip using the fabrication schedule outlined in Table 3.

TABLE 3. FABRICATION SCHEDULE APPLIED TO THE SOLID SOLUTION-DISPERSION ALLOYS

Rolling Stage	Rolling Temperature		Per Cent Reduction			Annealing Temperature		Annealing Time, min
	C	F	Per Pass	At Stage	Accumulated ^(a)	C	F	
Breakdown	1800	3270	20	30	30	--	--	--
Intermediate	1600	2910	20	30	50	--	--	--
Intermediate	1400	2550	15	40	70	1200	2190	30
Finish	1200	2190	15	50	85	--	--	--

(a) Calculated on the basis of a nominal initial thickness of 0.210 inch.

Rolling by this procedure was a moderately severe test of fabricability as it accomplished an 85 per cent total reduction to 0.030 to 0.035-inch strip while employing 20 and 15 per cent reductions per pass. Some general observations with respect to fabricability are presented as follows.

An unalloyed tungsten control bar (W-1) rolled along with the solid solution-dispersion alloys displayed excellent fabricability from initial breakdown rolling at 1800 C through finish rolling at 1200 C. No tendency was observed for this material to delaminate, split, or edge crack at any stage in the rolling process.

Finished strip of fair quality was also obtained from the W-5Re-(4, 5) ThO₂ and W-5Re-(4, 5, and 7) ZrO₂ alloys by this procedure. However, all of the alloy bars tended to alligator on breakdown rolling, an effect which was attributed to their rhenium inhomogeneity. Samples of each alloy are being examined metallographically to verify this suspicion. Nevertheless, the results of this experiment suggest that the range of workable oxide alloy content extends to at least 5 volume per cent of ThO₂ and ZrO₂ (2.7 and 1.5 weight per cent, respectively).

FUTURE WORK

Primary emphasis in the next quarter will be placed on experiments designed to select an appropriately doped base for use in further screening the W-5Re-(ThO₂ and ZrO₂) alloys.

Thus, evaluation of the present doped alloys will be continued as outlined in the body of this report. After fabrication, samples of each material will be evaluated to determine their softening and recrystallization behavior as well as their bend transition temperatures.

The most suitable base will then be used to compare the properties of ThO₂ and ZrO₂, at a level of 5 volume per cent, in combination with 5 per cent rhenium. Ultimately, either ThO₂ or ZrO₂, will be selected as the dispersoid addition to be used in a more extensive optimization of composition as well as fabrication procedures.

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- (1) Ratliff, J. L. , Maykuth, D. J. , Ogden, H. R. , and Jaffee, R. I. , "Development of a Ductile Tungsten Sheet Alloy", Summary Report on Contract NOw61-0677-C, May 26, 1962.
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- (3) Davis, G. L. , "The Recrystallization of Tungsten Wire", Metallurgia, 58, 177-184 (1958).

* * * *

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